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POLYMERS FOR SPACECRAFT HARDWARE --
MATERIALS SPECIFICATIONS AND ENGINEERING INFORMATION

Prepared for: Jet Propulsion Laboratory
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SRI Project ASD-5046

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Analyses and Instrumentation

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SCOPE

This report covers the work performed during the period August 10 to September 9, 1965 on "Polymers for Spacecraft Hardware-- Materials Specifications and Engineering Information, " SRI Project ASD-5046 under JPL Contract No. 950745.

The primary objectives of this program are to assist the Jet Propulsion Laboratory in the development and preparation of polymeric material specifications to be used in connection with JPL spacecraft, and to provide a study of the effects of simulated space environment on selected commercial polymeric products. The materials and products to be studied and the extent of work to be performed are specified by the JPL Cognizant Engineers.

The program is being conducted as two interrelated phases, running concurrently: The purpose of Phase I, Polymeric Materials Specifications, is to obtain quantitative values for parameters which may be used to assure the performance of a given batch of material in a spacecraft environment. The purpose of Phase II, Engineering Information, is to establish material limitations and to obtain detailed design information.

PHASE I - POLYMERIC MATERIALS SPECIFICATIONS

WORK PERFORMED

Work was continued with General Electric Silicone SE-555 (white) to establish a method of sample preparation which would provide optimum vacuum-weight-loss values. Samples were used in the form of large pieces, as finely-cut shreds, and as ground powders. Exposure times of 24, 48, 72, and 96 hours were selected in order to ascertain whether the weight-loss determinations could be made during an average work week. The results of these determinations are given in Table I. Since little difference in weight-loss values results from the various methods of preparation, the simplest method of handling is recommended for elastomer-type materials, i. e., pieces cut to maximum size to fit in the sample cells and be removed easily.

Some deviation in weight loss-values is attributable to weights being recorded at different times (within 1/2 hour). Although it is general knowledge that polymeric materials will adsorb moisture and air, even in a desiccator, after exposure to a vacuum-thermal environment, sufficient time must be allowed to permit the samples to reach an equilibrium temperature with that in the balance room, particularly when a micro or semimicro balance is employed. In view of the change in weight with time, illustrated below, an exact time for weighing samples after vacuum-thermal exposure is prescribed for specifications purposes; exactly one-half hour has been selected to provide maximum cooling and minimum weight increase.

Time lapse after removing samples from
vacuum-thermal environment and storing
in desiccator (SE-555)

Sample Wt., g

15 minutes

5.215907

30 minutes

5.216312

24 hours

5.217055

TABLE I EFFECT OF SAMPLE CONFIGURATION ON WEIGHT LOSS AT 125 °C AND 10 ⁻⁶ TORR FOR GENERAL ELECTRIC SILICONE SE-555 (WHITE)				
Treatment	Exposure, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
I*	24	2.315110	0.010750	0.46
	48	2.460585	0.010025	0.41
	72	2.377695	0.010035	0.42
	96	2.365140	0.010530	0.44
II*	24	1.570575	0.006270	0.40
	48	1.676655	0.007945	0.47
	72	1.730225	0.008160	0.47
	96	1.374530	0.006313	0.46
III*	24	1.368235	0.004835	0.35
	48	1.213260	0.006760	0.56
	72	1.264610	0.007355	0.58
	96	1.154275	0.006445	0.56

- * I Sample, as received, 3 strips of 2" x 1/2" x 1/16"
- * II Sample, as received, sliced with scalpel into pieces of about 1/2" x 1/16" x 1/64"
- * III Sample, as received, frozen with liquid nitrogen and ground in a mortar; dried under low vacuum at room temperature for 1/2 hour and stored in a desiccator until weighed.

Additionally, since the initial sample weight of polymeric materials is also dependent on ambient conditions, a standard procedure will be employed for ensuring reproducibility of results in any climate; samples are to be placed in a 50% relative-humidity environment (at 20-30 °C) for at least 24 hours prior to test.

The Silicone SE-555 apparently incurred a steady rate of weight-loss after the initial outgassing within the first 24 hours of exposure. Since the relatively low total weight-loss is well under the 1% often quoted as being permissible for space polymers, it seemed appropriate to suggest time exposures of 48 and 96 hours to establish weight loss and weight loss rate criteria for specifications purposes.

At the request of the JPL Cognizant Engineer, a series of RTV silicone potting compounds (General Electric) were selected for weight-loss determinations at 125 °C. The following materials were received in the cured state: RTV-11, RTV-560, RTV-615, and RTV-60. Duplicate samples of 2" x 1/2" were cut from the sheets, humidified as described above, and exposed to the thermal-vacuum environment in the equipment detailed in Interim Report No. 1; one sample of each was removed after 48 hours and the duplicate after 96 hours. All of these materials incurred weight-losses greater than 1%; the RTV-60 and RTV-615 had an apparent steady-state weight-loss increase of about 8% per hundred hours, but the value for RTV-11 was about 20% and for RTV-560 about 30%. In order to ascertain whether these "2-point" curves were valid, and whether the values were reproducible, fresh samples were prepared for exposure periods of 96 hours and 192 hours. As indicated in Table II, reproducibility of determinations at 96 hours varied from 0.00-0.12% abs. Additionally, when the resultant figures are plotted, the data indicate that steady-state weight loss increases are more nearly achieved after 96 hours for this class of materials. Perhaps the most reasonable approach for specifications purposes is to require that weight loss determinations be made in duplicate (not to vary more than $\pm 0.10\%$ abs) after 96 hours of exposure to the thermal-vacuum environment.

TABLE II
WEIGHT LOSS DATA FOR
GENERAL ELECTRIC RTV SILICONE
COMPOUNDS
(125 °C and 10⁻⁶ torr)

Sample	Exposure Time, Hrs.	S. Wt., g	Wt. Loss, g	Wt. Loss, %
RTV-60	48	3.893215	0.040140	1.03
	96	3.891950	0.042635	1.10
	96	3.870065	0.035610	0.92
	192	3.862725	0.036845	0.95
RTV-615	48	2.506210	0.036825	1.47
	96	2.601180	0.042615	1.64
	96	2.524705	0.041310	1.64
	192	2.661260	0.047050	1.77
RTV-11	48	3.017650	0.040165	1.33
	96	3.151675	0.046840	1.49
	96	2.893040	0.048050	1.66
	192	2.975525	0.050810	1.68
RTV-560	48	3.996970	0.137085	3.43
	96	3.635515	0.143520	3.95
	96	3.751525	0.139985	3.72
	192	3.901770	0.150150	3.85

A satisfactory procedure has been initiated for the determination of the amine values¹ for a series of Epon adhesives (Shell Chemical Company) and will be submitted in appropriate form for inclusion in the specifications requirements for epoxide adhesives. The amine values determined by this procedure were cross-checked by micro determinations for nitrogen; the results of these analyses are summarized in Table III.

<p style="text-align: center;">TABLE III</p> <p style="text-align: center;">DETERMINATIONS OF AMINE VALUES* FOR EPON RESINS AND CURING AGENTS</p>			
Epon No.	Amine Value	% N \approx Amine Value	% N, micro
931; Part B	970-972	24.3	27.4
901; Agent B-3	740-741	18.5	19.3
914	32-33	0.8**	3.6
422J	7.6-8.3	0.2**	1.6

*Amine value = milligrams of KOH equivalent to the basicity in 1 g of sample.

**An apparent value for the amine content.

Briefly, the procedure for the determination of amine values involves the titration of amine groups with perchloric acid in nonaqueous media (glacial acetic acid), using methyl violet indicator solution. In an effort to utilize standardized ASTM procedures, ASTM Method D 2073-62T (for fatty amines) was first investigated, but was found to have a weak and difficultly locatable end point, and low and inaccurate values were observed.

¹Welcher, F. J. (Editor), Standard Methods of Chemical Analysis, D. Van Nostrand Company, Inc., New York, 1963, Vol. II, Part A, p. 488.

FUTURE WORK

Vacuum weight loss determinations will be made on a continuing basis in order to establish limitations for specifications requirements for various polymeric materials. Investigations of physical and chemical properties of polymers will be conducted as required for incorporation into specifications requirements and test procedures.

PHASE II - ENGINEERING INFORMATION

WORK PERFORMED

Determinations of volatile condensable material (VCM value at 125/25 °C and 10^{-6} torr)* have been completed for Hypalon A-2211A-2718 and Vitons A-4411A-990 and -991 (DuPont). The results of these determinations are summarized in Tables IV, V, and VI and illustrated in Figures 1 and 2. Only a minor discoloration was observed on the polished-copper collecting-plugs for the Vitons; the deposition from Hypalon A-2211A-2718 was the same yellow-green coating noted from, Hypalon A-2211A-2717 in prior work at 143/25 °C.

As indicated in Tables IV and V, the greatest part of the weight lost from Vitons A-4411A-990 and -991 is attributable to noncondensable (at 25 °C) materials; a trend is also indicated toward reduction of condensable material which would imply the gradual pumping-off of light-molecular-weight VCM deposited initially on the cooled surfaces. In contrast, while the quantity of noncondensable gases released by the Hypalon A-2211A-2718 (Table VI) shows a smaller relative increase, the quantity of VCM indicates a marked increase with little evidence for the removal of initially-deposited VCM; the same general shape of curve was observed for Hypalon A-2211A-2717 at 143/25 °C, studied previously (see Interim Report No. 2, May 30, 1965, JPL Contract 950324).

Because of re-location and current modifications to the mass spectrometer used for identifying the substances released from polymeric materials, such analytical data are not available at this time.

In view of the large number of polymeric materials which must be evaluated, and the fact that plastics and laminates will be studied as well as elastomers, the VCM apparatus (described in the Interim

*VCM is defined as the weight of condensate obtainable at 25 °C in a given interval of time from a unit area of material of a given thickness maintained at 125 °C in a vacuum of at least 5×10^{-6} torr.

TABLE IV				
VCM DETERMINATION AT 125/25 °C AND 10 ⁻⁶ TORR FOR VITON A-4411A-990 (DuPont)				
Exposure Time, Hr.	S. Wt., g	Weight Loss, %		VCM Value 125/25 °C, mg/in ²
		Condensable	Noncondensable	
24	6.025544	0.05	0.21	1.43
48	6.428760	0.02	0.19	0.49
96	6.154627	0.03	0.20	0.73
300	6.229930	0.02	0.23	0.51

TABLE V				
VCM DETERMINATION AT 125/25 °C AND 10 ⁻⁶ TORR FOR VITON A-4411A-991 (DuPont) .				
Exposure Time, Hr.	S. Wt., g	Weight Loss, %		VCM Value 125/25 °C, mg/in ²
		Condensable	Noncondensable	
24	7.019872	0.03	0.07	1.03
48	6.760318	0.03	0.19	1.11
96	6.942017	0.02	0.27	0.57
300	6.883503	0.01	0.07	0.41

TABLE VI				
VCM DETERMINATION AT 125/25 °C AND 10 ⁻⁶ TORR FOR HYPALON A-2211A-2718 (DuPont)				
Exposure Time, Hr.	S. Wt., g	Weight Loss, %		VCM Value 125/25 °C, mg/in ²
		Condensable	Noncondensable	
24	4.717015	0.18	0.57	3.58
48	4.767385	0.33	0.60	6.88
96	4.823076	0.43	0.64	9.05
300	4.797965	1.06	0.64	22.03

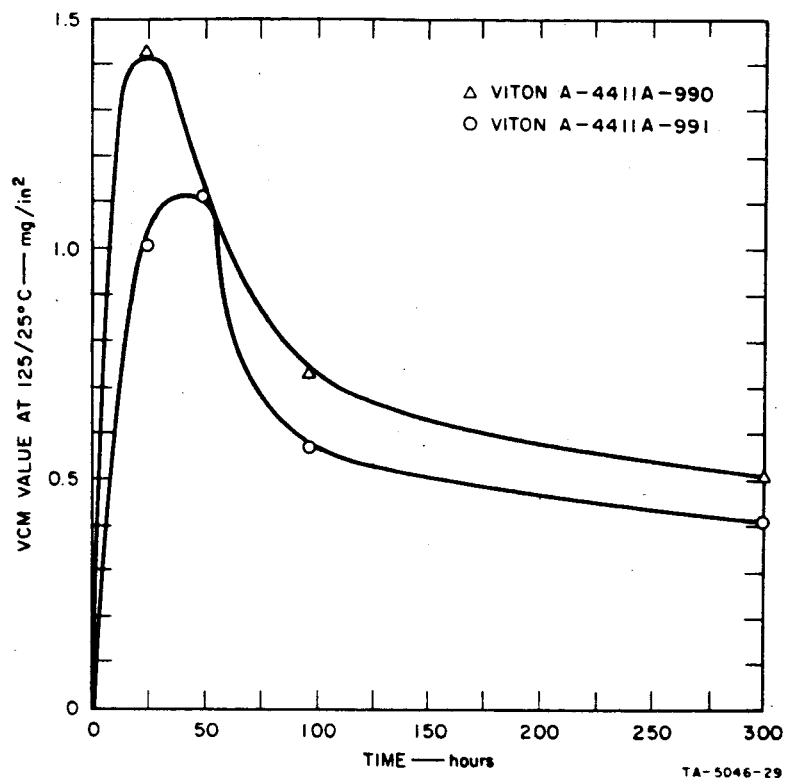


FIG. 1 VCM CURVES FOR VITON SAMPLES AT 125/25°C

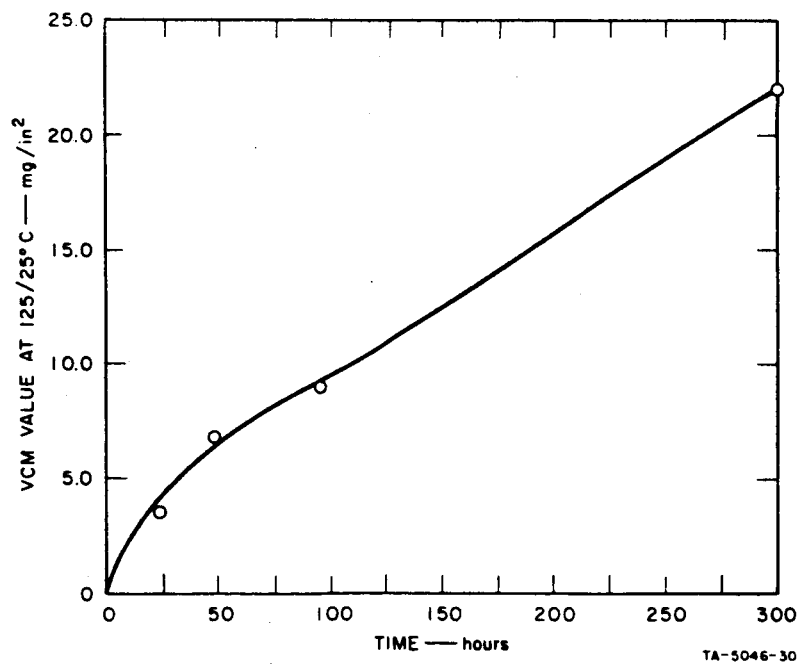


FIG. 2 VCM CURVE FOR HYPALON A-2211A-2718 AT 125/25°C

Report previously cited) has been disassembled, and construction on an improved form of VCM units is well underway. The new units will provide for simultaneous VCM determinations for 10 to 12 materials, and may best be described as individual "clam-shells," with appropriate supports to accommodate any type of sample. The clam-shells have been spun from copper, and at the present the heating elements are being affixed. Appropriate design drawings and illustrations will be published when the design, construction and operation are finalized.

FUTURE WORK

Work will continue toward the completion and actual operation of the improved VCM units.

It is anticipated that work will commence on the identification of released substances via mass spectroscopy and the characterization by infrared techniques of polymeric materials before and after vacuum-thermal exposure.